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IS 14157 (1994): Simazine, Technical [FAD 1: Pesticides and Pesticides Residue Analysis]

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IS 14157 : 1994
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भारतीय मानक

सिमाज़िन, तकनीकी — विशिष्टि

Indian Standard

SIMAZINE, TECHNICAL — SPECIFICATION

UDC 632.954 SIM

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BUREAU OF INDIAN STANDARDS
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI 110 002

June 1994

Price Group 2

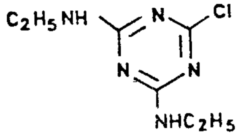
Pesticides Sectional Committee, FAD 1

FOREWORD

This Indian Standard was adopted by the Bureau of Indian Standards, after the draft finalized by the Pesticides Sectional Committee had been approved by the Food and Agriculture Division Council.

Simazine, technical is employed in the preparation of herbicidal formulations.

Simazine is the accepted common name by the International Organization for Standardization (ISO) for 2-chloro-4, 6-bis-(ethylamino)-1, 3, 5-triazine. The empirical and structural formulae and molecular mass of simazine are given below:

Empirical Formula	*Structural Formula*	*Molecular Mass*
$C_7H_{12}ClN_5$		201.7

In the preparation of this standard, due consideration has been given to the provisions of the *Insecticides Act*, 1988 and the Rules framed thereunder and *Standards of Weights and Measures (Packaged Commodities), Rules* 1977. However, this standard is subject to the restrictions imposed under the *Act* and Rules wherever applicable.

For the purpose of deciding whether a particular requirement of this standard is complied with the final value, observed, or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS 2 : 1960 'Rules for rounding off numerical values (*revised*)'. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

Indian Standard

SIMAZINE, TECHNICAL — SPECIFICATION

1 SCOPE

This standard prescribes the requirements and the methods of sampling and test for simazine, technical.

2 REFERENCES

The following Indian Standards are necessary adjuncts to this standard:

IS No.	*Title*
1070 : 1992	Reagent grade water (*third revision*)
6940 : 1982	Method of test for pesticides and their formulations (*first revision)*
8069 : 1989	High density polyethylene (HDPE) woven sacks for packing pesticides (*second revision*)
8190 (Part 1): 1988	Requirement for packing of pesticides: Part 1 Solid pesticides (*second revision*)
10946 : 1984	Method of sampling for technical grade pesticides.

3 REQUIREMENTS

3.1 Description

The material shall be in the form of white powder, free from extraneous impurities and/or added modifying agent(s).

3.2 The material shall also comply with the requirements given in Table 1.

4 PACKING

The material shall be packed in mild steel/HDPE woven sacks laminated with 0·025 mm LDPE [*see* IS 8069 (Part 1) : 1981]. The general requirements given in IS 8190 (Part 1) : 1988 shall also be followed.

5 MARKING

5.1 The container shall bear legibly and indelibly the following information in addition to any other information as is necessary under the *Insecticides Act*, 1968 and Rules framed thereunder:

a) Name of the material;

b) Name of the manufacturer;

c) Batch number;

d) Date of manufacture;

e) Date of expiry;

f) Simazine content, percent (*m/m*);

g) Net mass of the contents;

h) Cautionary notice as worded in the *Insecticides Act*, 1968 and Rules framed thereunder; and

j) Any other information(s) required under the *Standards of Weights and Measures (Packaged Commodities) Rules*, 1977.

5.2 BIS Certification Marking

The product may also be marked with Standard Mark.

Table 1 Requirements for Simazine, Technical
(*Clauses* 3.2 *and* 7.1)

Sl No.	Characteristic	Requirement	Method of Test, Ref to	
			Annex of This Standard	Cl No. of IS 6940 : 1982
(1)	(2)	(3)	(4)	(5)
i)	Simazine content, percent by mass, (*m/m*) *Min*	95·0	A	—
ii)	Moisture content, percent by mass, (*m/m*) *Max*	2·0	—	4.1
iii)	Ionic chloride content (as NaCl), percent by mass, (*m/m*) *Max*	2·0	B	—

5.2.1 The use of the Standard Mark is governed by the provisions of Bureau of Indian Standards Act, 1986 and the Rules and Regulations made thereunder. The details of conditions under which the licence for the use of Standard Mark may be granted to manufacturers or producers may be obtained from the Bureau of Indian Standards.

6 SAMPLING

Representative samples of the material shall be drawn according to IS 10946 : 1984.

7 TESTS

7.1 Tests shall be carried out by the methods referred to in col 4 and 5 of the Table 1.

7.2 Quality of Reagents

Unless specified otherwise, pure chemicals and distilled water (*see* IS 1070 : 1992) shall be employed in tests.

NOTE — 'Pure chemicals' shall mean chemicals that do not contain inpurities which affect the results of analysis.

ANNEX A

[*Clause* 3.2, and *Table* 1, *item* (i)]

DETERMINATION OF SIMAZINE CONTENT

A-0 GENERAL

Either of the two methods, namely high performance liquid chromatography (HPLC) and alkaline hydrolysis method may be used for the determination of simazine content. However, the HPLC method shall be the referee method.

A-1 HPLC METHOD

A-1.1 Principle

The sample is diluted to appropriate volume with methyl alcohol and simazine content is determined using diphenyl oxide as internal standard, by high performance liquid chromatography.

A-1.2 Apparatus

A-1.2.1 *Syringe* — 20 μl capacity

A-1.2.2 *High Performance Liquid Chromatograph (HPLC Unit)*

An HPLC unit equipped with an ultra-violet detector and coupled to a printer-plotter *cum* integrator. The operative parameters are suggested below, which can be changed in any other equipment, provided standardization is done:

Column — Stainless steel (Zerbax ODS or equivalent), 25 cm length × 4.6 mm ID;
Detector — UV (254 nm)
Solvent System — Methyl alcohol
Solvent Flow Rate — 1 ml/min
Chart Speed — 0·5 cm/min
Sample size — 20 μl

A-1.3 Reagents

A-1.3.1 *Internal Standard*—Diphenyl oxide, AR grade

A-1.3.2 *Methyl Alcohol*— Spectroscopic grade

A-1.3.3 *Simazine Reference Standard*—of known purity

A-1.4 Procedure

A-1.4.1 *Preparation of Internal Standard Solution*

Weigh accurately 1·0 diphenyl oxide in a 100-ml volumetric flask and make up the volume to the mark with methyl alcohol. This will give a solution containing 10 mg/ml of diphenyl oxide.

A-1.4.2 *Preparation of Standard Solution*

Weigh accurately 0·1 g of simazine reference standard in a 100-ml volumetric flask. Add 10 ml of internal standard solution and make up the volume to the mark with methyl alcohol. This solution will contain 0·1 mg/ml of simazine.

A-1.4.3 *Preparation of Sample Solution*

Weigh accurately a quantity of sample to contain 0·1 g of simazine into a 100-ml volumetric flask. Add 10 ml of the internal standard solution and make up to the mark with methyl alcohol.

A-1.4.4 *Estimation*

Inject 20 μl of the standard solution into the gas chromatograph followed by the sample solution. Measure the peak area of the internal standard, reference standard and sample. Compute the percentage of simazine in the sample.

A-1.5 *Calculation*

$$\text{Simazine content, percent by mass} = \frac{M_1 \times A_1 \times A_3 \times P}{M_2 \times A_2 \times A_4}$$

where

M_1 = mass, in g, of reference standard simazine;
M_2 = mass, in g, of sample taken for the test;
A_1 = peak area of internal standard in the standard solution injected;

A_2 = peak area of simazine in the standard solution injected;

A_3 = peak area of simazine in the sample solution injected;

A_4 = peak area of internal standard in the sample solution injected; and

P = percent purity of simazine reference standard.

A-2 ALKALINE HYDROLYSIS METHOD

A-2.1 Principle

The chlorine atom in simazine is converted to ionic chloride. The total ionic chloride thus formed is estimated titrimetrically by Volhard's procedure. The determined chloride after multiplying by appropriate conversion factor gives the simazine content.

A-2.2 Reagents

A-2.2.1 *Alcoholic Potassium Hydroxide Solution* — 1 N

Dissolve 56 g of potassium hydroxide in 1 000 ml of anhydrous ethanol.

A-2.2.2 *Nitric Acid* — 1:1 (*v* / *v*)

A-2.2.3 *Silver Nitrate Solution* — 0·1 N

A-2.2.4 *Standard Potassium Thiocyanate Solution* — 0·1N

A-2.2.5 *Phenolphthalein Indicator Solution* — 1 percent in 96 percent ethyl alcohol (*v* / *v*).

A-2.2.6 *Ferric Alum Indicator*

A-2.3 Procedure

A-2.3.1 Weigh accurately about 1·0 g of technical sample and transfer quantitatively into a 250-ml Erlenmeyer flask with ground glass joint. Add 50 ml alcoholic potassium hydroxide solution and reflux gently for 30 minutes. Cool, rinse the condensor with 50 ml water. Neutralise with nitric acid using phenolphthalein as indicator. Add 10 ml in excess. Add 25 ml of silver nitrate solution accurately with a pipette and back titrate the excess against standard potassium thiocyanate solution using ferric alum as indicator.

A-2.3.2 Carry out a blank determination with same weight of the material and back titrating against standard potassium thiocyanate solution as above to make correction for ionic chloride.

A-2.4 Calculation

Simazine content, percent by mass =

$$\frac{20{\cdot}17 \times (V_2 - V_1) \times N}{M} - \text{Ionic chloride content, percent by mass}$$

V_1 = volume, in ml, of standard potassium thiocyanate solution consumed for the titration for sample;

V_2 = volume, in ml, standard potassium thiocyanate solution required for blank determination;

N = normality of standard potassium thiocyanate solution; and

M = mass, in g, of the sample taken for the test.

ANNEX B

[*Clause* 3.2, and *Table* 1, *Item* (iii)]

DETERMINATION OF IONIC CHLORIDE CONTENT

B-1 REAGENTS

B-1.1 Dilute Nitric Acid — 10 percent (v/v)

B-1.2 Silver Nitrate Solution — 0·1 N

B-1.3 Standard Potassium Thiocyanate Solution — 0·1 N

B-1.4 Ferric Alum Indicator

B-2 PROCEDURE

Weigh accurately about 1 g of technical sample and transfer quantitatively into a 250-ml Erlenmeyer flask. Add 50 ml water and 10 ml dilute nitric acid. Then add 25 ml of silver nitrate solution with a pipette and add 2 ml of the ferric alum indicator solution. Titrate against standard potassium thiocyanate solution till faint red colour is obtained. Carry out a blank titration.

B-3 CALCULATION

Ionic Chloride (as NaCl) content, percent by mass =

$$\frac{5.846 \times (V_1 - V_2) \times N}{M}$$

where

V_1 = volume, in ml, of standard potassium thiocyanate solution consumed for the titration for sample;

V_2 = volume, in ml, of standard potassium thiocyanate solution required for blank determination;

N = normality of standard potassium thiocyanate solution; and

M = mass, in g, of the sample taken for the test.

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